

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE****IN RE APPLICATION OF MICHAEL A. LEVY****SERIAL NO. 10/563,207****FILED: JUNE 30, 2006****PRIORITY: FEBRUARY 2, 2003****FOR: IN-SITU TREATMENT OF PYRIDINE 2,3-DICARBOXYLIC ACID ESTERS WITH AN OXIDIZING AGENT****DECLARATION**

I, David A. Cortes, a Doctor of Chemistry, a citizen of the United States of America and residing at 4502 Marlon Court, Quincy, IL 62305, USA, declare as follows:

I am a fully trained chemist, having studied chemistry at the Columbia University in New York, NY from 1976 to 1981;

I was awarded my Ph.D. by the latter university in 1981;

I was a post-doctoral fellow at the Princeton University in Princeton, NJ from 1981 to 1983;

Since 1983, when I joined American Cyanamid, later acquired in 2000 by BASF Corporation of Florham Park 07932, NJ, USA, I have been engaged in the synthesis of organic compounds;

I am fully conversant with the technical area to which application No. 10/563,207 pertains;

I have read the application and studied the application file, in particular the Office Action dated 03/24/2008, and the prior art referenced therein, and I am therefore also well acquainted with the invention, which is disclosed and claimed in application No. 10/563,207;

It is my understanding the Examiner questions whether the data and the example, which are described in the application, are representative of the diester structures and oxidizing agents within the scope of the claims of the application, which had not been included in the example described in the application.

The following tests and results show, in my opinion, that the claimed method for in-situ removal of impurities is not limited to the diester structures and oxidizing agents which were employed in the example described in the application, and that the data set forth in the application can reasonably be extended as being representative of the properties of all diester structures and oxidizing agents which are defined by the claims.

The supplemental examples A to F were carried out under my supervision in accordance with the standardized procedures described on paragraphs [0015] to [0030] of the published application. I have reviewed the test protocols and based on my review and knowledge I consider those data to be fully reliable.

### Example A

#### 5-Ethylpyridine-2,3-dicarboxylic acid crude diester / Hydrogen peroxide

A saponification mixture of 5-Ethylpyridine-2,3-dicarboxylic acid crude diester (100 grams), water (103 grams), and 50% NaOH (76 grams) was heated to 100°C. The alcohol distillate by-product of the saponification mixture was collected (33 grams). This was followed by the slow addition of 30 grams of 35% H<sub>2</sub>O<sub>2</sub> to the diacid salt solution over 1 hour, maintaining the temperature at 95°C. The solution was initially black in color. Addition of the H<sub>2</sub>O<sub>2</sub> caused foaming. Foaming was markedly reduced and reaction color changed to light amber as the addition of the H<sub>2</sub>O<sub>2</sub> proceeded. After addition of all H<sub>2</sub>O<sub>2</sub>, the solution was stirred for 2 hours while maintaining a temperature of 95°C. This was followed by testing for residual peroxide using KI/starch paper, which tested negative. Water was then added (67 grams) to the oxidized solution.

The product was isolated by precipitation and the filtercake was washed with water (50 grams). The wet filtercake had a mass of 76.3 grams and the mother liquor (310 grams) containing 4% diacid was recycled. The resulting off-white filtercake was dried overnight. The dried recovered diacid product had a mass of 61.0 grams at 98.9% purity.

### Example B

#### 5-Methylpyridine-2,3-dicarboxylic acid crude diester / Hydrogen peroxide

A saponification mixture of 5-Methylpyridine-2,3-dicarboxylic acid crude diester (88 grams), water (103 grams), and 50% NaOH (76 grams) was heated to 100°C. The alcohol distillate by-product of the saponification mixture was collected but not weighed. At distillate collection completion, water (100 grams) was added to the reaction solution. This was followed by the slow addition of 7 grams of 35% H<sub>2</sub>O<sub>2</sub> to the diacid salt solution over 1 hour, maintaining the temperature at 95°C. The solution was initially black in color. Reaction color was markedly reduced and changed to light amber as the addition of the H<sub>2</sub>O<sub>2</sub> proceeded. After addition of all H<sub>2</sub>O<sub>2</sub>, the solution was stirred for 15 minutes while maintaining a temperature of 95°C. This was followed by testing for residual peroxide using KI/starch paper, which tested negative. Water (75 grams) was then added to the oxidized solution.

The product was isolated by precipitation and the filtercake was washed with water (100 grams). The wet filtercake had a mass of 89 grams and the mother liquor (466 grams) contained 0.5% diacid. The resulting off-white filtercake was dried overnight. The dried recovered diacid product had a mass of 62.6 grams at 99% purity.

**Example C****5-Methoxymethylpyridine-2,3-dicarboxylic acid Diester / Hydrogen peroxide**

Water (40 grams) was added to a saponified solution of 5-Methoxy methylpyridine-2,3-dicarboxylic acid Diester (367 grams) and heated to 85°C. This was followed by the quick addition of 10.2 grams of 35% H<sub>2</sub>O<sub>2</sub> while maintaining the temperature at 85°C. The solution was initially black in color. Reaction color was markedly reduced and changed to light amber after the addition of H<sub>2</sub>O<sub>2</sub>. After addition of all H<sub>2</sub>O<sub>2</sub>, the solution was stirred for 30 minutes while maintaining a temperature of 85°C. This was followed by testing for residual peroxide using KI/starch paper, which tested negative.

The product was isolated by precipitation and the filtercake was washed with water (50 grams). The wet filtercake had a mass of 44.2 grams and the mother liquor (448 grams) containing 4.6% diacid was recycled. The resulting light beige filtercake was dried overnight. The dried recovered diacid product had a mass of 26 grams at 96% purity.

**Example D****5-Methoxymethylpyridine-2,3-dicarboxylic acid Diester / Sodium hypochlorite**

To a saponified solution of 5-Methoxy methylpyridine-2,3-dicarboxylic acid Diester (367 grams) was added by the slow addition, 148.9 grams of 5.25% Na(OCl) (bleach) at ambient temperature. The solution was initially black in color. Reaction color was noticeably reduced and changed to light amber after the addition of Na(OCl). After addition of all Na(OCl), the solution was stirred for 60 minutes while maintaining ambient temperature.

**Example E****5-Methoxymethylpyridine-2,3-dicarboxylic acid Diester / Calcium hypochlorite**

Water (40 grams) was added to a saponified solution of 5-Methoxy methylpyridine-2,3-dicarboxylic acid Diester (367 grams). This was followed by the slow addition of 23.1 grams of 65% Ca(OCl)<sub>2</sub> (bleach powder) at ambient temperature. The solution was initially black in color. Reaction color was markedly reduced and changed to light amber after the addition of Ca(OCl)<sub>2</sub>. After addition of all Ca(OCl)<sub>2</sub>, the solution was stirred for 60 minutes while maintaining ambient temperature.

## Example F

### 5-Methoxymethylpyridine-2,3-dicarboxylic acid Diester / Calcium hypochlorite

Water (40 grams) was added to a saponified solution of 5-Methoxy methylpyridine-2,3-dicarboxylic acid Diester (367 grams). This was followed by the slow addition of 0.1 grams of 65%  $\text{Ca}(\text{OCl})_2$  (bleach powder) at ambient temperature. The solution was initially black in color. Reaction color was markedly reduced and changed to light amber after the addition of  $\text{Ca}(\text{OCl})_2$ . After addition of all  $\text{Ca}(\text{OCl})_2$ , the solution was stirred for 60 minutes while maintaining ambient temperature.

Together with the Example 1 and the data which are set forth on paragraph [0016] to [0030] of the application, the foregoing supplemental examples corroborate that diester structures and oxidizing agents disclosed and claimed in the application are not limited to the investigated compounds in Example 1. The diester structures and oxidizing agents within the boundaries set by the claims are equally useful for the method for the in-situ removal of impurities according to the claims.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 101 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at Palmyra, Missouri 63461, this 13 day of May, 2008.



Signature of Declarant  
David A. Cortes